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CONCERNING A FILING UNDER 35 U.S.C. 371**

U.S. APPLICATION NO. (If known, see 37 CFR 1.5)

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INTERNATIONAL APPLICATION NO.
PCT/DK99/00559

INTERNATIONAL FILING DATE
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TITLE OF INVENTION

METHOD FOR THE PRODUCTION OF FEED PELLETS, MIX OF A PREMIX AND VITAMIN PREMIX

APPLICANT(S) FOR DO/EO/US

Victor DUER

Applicant(s) herewith submits to the United States Designated/Elected Office (DO/EO/US) the following items and other information

1. ☒ This is a **FIRST** submission of items concerning a filing under 35 U.S.C. 371.
2. ☐ This is a **SECOND** or **SUBSEQUENT** submission of items concerning a filing under 35 U.S.C. 371.
3. ☒ This is an express request to begin national examination procedures (35 U.S.C. 371(f)) immediately rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39(1).
4. ☒ A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.
5. ☒ A copy of the International Application as filed (35 U.S.C. 371(c)(2))
 - a. ☐ is transmitted herewith (required only if not transmitted by the International Bureau).
 - b. ☒ has been transmitted by the International Bureau.
 - c. ☐ is not required, as the application was filed in the United States Receiving Office (RO/US)
6. ☒ A translation of the International Application into English (35 U.S.C. 371(c)(2)).
7. ☒ Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3))
 - a. ☐ are transmitted herewith (required only if not transmitted by the International Bureau).
 - b. ☐ have been transmitted by the International Bureau.
 - c. ☐ have not been made; however, the time limit for making such amendments has NOT expired.
 - d. ☒ have not been made and will not be made.
8. ☐ A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).
9. ☐ An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4))
10. ☒ A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).

Items 11. to 16. below concern other document(s) or information included:

11. ☒ An Information Disclosure Statement under 37 CFR 1.97 and 1.98.
12. ☐ An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.
13. ☐ A **FIRST** preliminary amendment.
☐ A **SECOND** or **SUBSEQUENT** preliminary amendment.
14. ☐ A substitute specification and marked-up version.
15. ☐ A change of power of attorney and/or address letter.
16. ☒ Other items or information: International Search Report, Form PCT/RO/101, International Preliminary Examination Report

Express Mail No.:EL243104167US

U.S. APPLICATION NO. 01/831243 (See 37 CFR 1.5)INTERNATIONAL APPLICATION NO.
PCT/DK99/00559ATTORNEY'S DOCKET NUMBER
12085/117. ☒ The following fees are submitted:**Basic National Fee (37 CFR 1.492(a)(1)-(5)):**

Search Report has been prepared by the EPO or JPO \$860.00

International preliminary examination fee paid to USPTO (37 CFR 1.482) \$690.00

No international preliminary examination fee paid to USPTO (37 CFR 1.482) but
international search fee paid to USPTO (37 CFR 1.445(a)(2)) \$710.00Neither international preliminary examination fee (37 CFR 1.482) nor international
search fee (37 CFR 1.445(a)(2)) paid to USPTO \$1,000.00International preliminary examination fee paid to USPTO (37 CFR 1.482) and all
claims satisfied provisions of PCT Article 33(2)-(4) \$100.00

CALCULATIONS | PTO USE ONLY

ENTER APPROPRIATE BASIC FEE AMOUNT =

\$ 860

Surcharge of \$130.00 for furnishing the oath or declaration later than ☐ 20 ☐ 30 months
from the earliest claimed priority date (37 CFR 1.492(e)).

\$

| Claims | Number Filed | Number Extra | Rate | | |
|---|--------------|--------------|------------|-------|--|
| Total Claims | 14 - 20 = | 0 | X \$18.00 | \$0 | |
| Independent Claims | 2 - 3 = | 0 | X \$80.00 | \$0 | |
| Multiple dependent claim(s) (if applicable) | | | + \$270.00 | \$270 | |

TOTAL OF ABOVE CALCULATIONS =

\$1130

Reduction by 1/2 for filing by small entity, if applicable. Verified Small Entity statement must
also be filed. (Note 37 CFR 1.9, 1.27, 1.28).

\$

SUBTOTAL =

\$1130

Processing fee of \$130.00 for furnishing the English translation later the ☐ 20 ☐ 30
months from the earliest claimed priority date (37 CFR 1.492(f)).

+

\$

TOTAL NATIONAL FEE =

\$1130

Fee for recording the enclosed assignment (37 CFR 1.21(h)). The assignment must be
accompanied by an appropriate cover sheet (37 CFR 3.28, 3.31). \$40.00 per property

+

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TOTAL FEES ENCLOSED =

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- a. ☐ A check in the amount of \$_____ to cover the above fees is enclosed.
- b. ☒ Please charge my Deposit Account No. 11-0600 in the amount of \$1130.00 to cover the above fees. A duplicate copy of this sheet is enclosed.
- c. ☒ The Commissioner is hereby authorized to charge any additional fees which may be required, or credit any overpayment to Deposit Account No. 11-0600. A duplicate copy of this sheet is enclosed.

NOTE: Where an appropriate time limit under 37 CFR 1.494 or 1.495 has not been met, a petition to revive (37 CFR 1.137(a) or (b)) must be filed and granted to restore the application to pending status.SEND ALL CORRESPONDENCE TO:
Kenyon & Kenyon

SIGNATURE

Christine M. Wilkes, Reg. No. 37,967

NAME

DATE



26646

PATENT TRADEMARK OFFICE

METHOD FOR THE PRODUCTION OF FEED PELLETS. MIX OF A
PREMIX AND VITAMIN PREMIX.

5 The invention concerns a method for the production of animal feed pellets with the addition of a premix.

The invention also concerns a method for the mixing of a vitamin premix comprising fat/oil-soluble vitamins.

10 Finally, the invention comprises a vitamin premix comprising soluble vitamins.

15 It is known from EP-A-682874 to add vitamins to animal feeds, said vitamins existing in a fat or oil solution, by which product and by which method there should arise a smaller risk of the decomposition of the vitamin admixture. However, the publication does not disclose a method for the addition of the total physiological vitamin requirement which is necessary in connection with animal feeds, and the publication does not either state any method whereby this admixture of vitamins takes place under conditions
20 where damage and decomposition of the individual vitamins, whether both fat and water soluble, is minimised to the greatest possible degree.

25 It is the object of the present invention to provide a method whereby the above-mentioned problems are overcome, and whereby the percentage of recovery for the added vitamins is very high, whereby overdosing is avoided, and whereby it is possible to effect a spraying of liquid premixes on the feed pellets, so that investments in larger plants for the handling of premixes following the dry admixture method is made superfluous.

30 It is also the object by this method to ensure that the processing and storage stability is optimised, so that the least possible decomposition of

the vitamins occurs after these have been sprayed on to the industrially-produced feeds.

5 This object is achieved with a method of the kind disclosed in the preamble, and also where the premix is a vitamin premix comprising fat/oil and water-soluble vitamins, where the surface of the feed pellets is sprayed with the vitamin premix, and where the feed pellets have been subjected to cooling-before the spraying.

10 By carrying out a spraying of the feed pellets when these are produced, and by also carrying out a suitable cooling to a maximum of 50°C and an optimum temperature level of 25-35°C, it is possible to effect a subsequent spraying of the surface of the pellets with the vitamin premix, which comprises both fat/oil- and water-soluble vitamins, whereby it is ensured that
15 the feed pellets receive an optimum content of vitamins for the relevant species of animals, whether these be pigs or chickens etc. By this method, a far higher recovery of the added vitamins is achieved than with a corresponding dosing via a dried vitamin premix.

20 The substantially improved recovery percentage for the vitamins after spraying is of importance for the animals' vitamin supply, and is thus also significant in relation to the feed control authorities. Consequently, it is important that the dosing of vitamins by liquid spraying can be reduced without reducing the animals' supply of added vitamins.

25 By providing a method according to the invention and as further disclosed in claim 2, it is achieved that the feed pellets are stored at a relatively low concentration of oxygen, whereby the decomposition is minimised and the storage stability is optimised. A storage time of 2-6 weeks, which is a
30 relatively short but nevertheless a realistic storage time, thus results in the vitamin stability and the recovery being surprisingly great.

By providing a method according to the invention and as further disclosed in claim 3, the possibility is established for a regulation of the finish-produced feed pellets with the vitamin premix with which they are sprayed, all depending on whichever animal may be involved. In other words, this means that where the feed is to be used, for example, for chickens, the pellets can be sprayed with vitamins comprising the C vitamin.

By providing a method according to the invention, and as further disclosed in claim 4, a good spraying on the individual pellets is achieved, so that any section of the pellets will give rise to substantially the same amount of concentration of the premix with which they are sprayed.

By providing a method according to the invention and as further disclosed in claim 5, it is achieved that the feed pellets also come to comprise the minerals which may be relevant for the individual animals.

By providing a method according to the invention and as further disclosed in claim 6, the possibility is achieved of adding further nutrients such as lysine, methionine, threonine, leucine and isoleucine.

By providing a method according to the invention and as further disclosed in claim 7, the possibility is achieved for the addition of enzymes such as carbohydrate- and protein-spliced enzymes.

By providing a method according to the invention and as further disclosed in claim 8, a saving is achieved in the amount of added phosphor, and which otherwise means that the selected B vitamin should predominantly be riboflavin and not sodium riboflavin phosphate.

As disclosed in the preamble, the invention also concerns a method for the mixing of the actual vitamin premix which is used during the spraying, and

where the premix also includes water-soluble vitamins, that the water phase comprises propylene glycol and EDTA and nicotinamide, after which there is subsequently added a B₂ vitamin such as riboflavin and sodium hydroxide (NaOH).

5

With such a premix comprising the above-mentioned components, the possibility is provided of dissolving the riboflavin, which is important in that riboflavin is not only advantageous from the point of view of cost, but also necessary to use as B₂ vitamin if phytase enzyme is to be added later.

10

By providing a method according to the invention and as further disclosed in claim 10, the possibility is provided of being able to add greater amounts of B₂ vitamin such as riboflavin.

15

By providing a method according to the invention and as further disclosed in claim 11, a reduction in the pH is achieved, so that the added riboflavin or sodium riboflavin phosphate is not ruined by the high pH which is established by the addition of sodium hydroxide (NaOH).

20

By providing a method according to the invention and as further disclosed in claim 12, an expedient composition of the oil phase is achieved.

25

By providing a method according to the invention and as further disclosed in claim 13, a homogenous substance of the premix is achieved during stirring.

30

By providing a method according to the invention and as further disclosed in claim 14, it is achieved that the amount of phosphate secreted by the animals is reduced. Moreover, the solubility of the riboflavin is ensured due to the presence of urea.

The temperature of 20-30° in the premix can be achieved by the addition of cold water before the addition of the phytase.

The invention also concerns a vitamin premix such as that disclosed in claim 15.

The invention will be explained in more detail with reference to the following example:

10

LIQUID VITAMINS

| <i>Code</i> | <i>Raw material</i> | <i>Weight in grams</i> | |
|-------------|--|------------------------|---------------|
| 01-A | Vitamin-A-acetate 2.5 mill. IU/g | 0.930 | Oil phase |
| 02-A | Vitamin-D ₃ -oil 4.0 mill. IU/g | 0.060 | - |
| 03-A | Vitamin-E-oil 97% | 39.950 | - |
| 04-A | Ethoxyquin | 2.000 | - |
| 05-A | Bredol 694 | 50.000 | - |
| | | | |
| 01-B | Choline chloride 75% solution* | 0 | Water phase I |
| 02-B | Vitamin-B ₁₂ 2% solution | 0.610 | - |
| 03-B | Panthenol | 5.100 | - |
| 04-B | Biotin 2% to liquid vitamins | 1.040 | - |
| 05-B | Pyridoxine hydrochloride | 1.240 | - |
| 06-B | Sodium riboflavin phosphate | 0.590 | - |
| 07-B | Sodium hydroxide solution 34° Bé | 2.600 | |
| 08-B | Riboflavin 97% | 0.590 | - |
| 09-B | Hydrochloric acid 10% | 2.800 | - |
| 09-B | Folinic acid 100%* | 0 | - |

| <i>Code</i> | <i>Raw material</i> | <i>Weight in grams</i> | |
|-------------|--|------------------------|----------------|
| 10-B | Urea 46 (Carbamide) | 50.000 | - |
| 11-B | Nicotinamide (=niacinamide) | 9.050 | - |
| 12-B | Potassium sorbate (preservative agent) | 2.000 | - |
| 13-B | Tetracemindinsodium (complex binder) | 2.000 | - |
| 14-B | Propylene glycol (dissolution- promoting and preservative agent) | 100.000 | - |
| 15-B | Water | 666.550 | - |
| | | | |
| 01-C | Phytase solution 5000 FTU/g | 60.000 | Water phase II |
| 02-C | Thiamine hydrochloride | 0.990 | - |
| 03-C | Menadione sodium bisulphate 100% | 1.900 | - |
| | Total – grams: | 1000.000 | |

* relevant for poultry.

Before the 07 base is added, the pH is approx. 6, after which the base ad-
 5 dition results in a pH increase to approx. 10-11, which is necessary in order
 for the riboflavin (08) to be dissolved. Component 09 is then added to effect
 a reduction of the pH to around 8, in that the substance 08 is ruined by a
 pH which is too high. By carrying out a mixing as described, it is achieved
 that all vitamins are present in the desired concentrations, and without
 10 destruction of these taking place.

Generally, the carbamide weight percentage interval of the total amount will
 lie in the interval 2-10%, mainly around 5%.

If the sodium riboflavin phosphate is omitted, the necessary amount of carbamide in the example will increase to 50-60 grams in order to ensure the solubility of the riboflavin.

- 5 The solution itself consisting of both water-soluble as well as oil-soluble vitamins is subsequently sprayed on the feed pellets for animals. This spraying can be effected before the pellets are stored in silos, or spraying can be effected when the pellets are loaded into transport containers. Both solutions enable a differentiation to be made between the different animals,
- 10 e.g. pigs vs. chickens, and differentiation in accordance with customer requirements.

- It has shown surprisingly that when this spraying of the vitamins on the pellets is carried out, no destruction or precipitation of the vitamins occurs,
- 15 which means that the pellets can be considered to be particularly stable. The reason why no destruction occurs can possibly be that the pellets are stored in silos and containers in which there is a low percentage of oxygen.

Production principle

- 20 The final preparation consists of an oil phase comprising the so-called fat-soluble vitamins mixed up with an adjusted amount of a suitable solubility product – here, use is made of Bredol 694. This mixture, preheated to 55-65°C, is poured/pumped in an adjusted flow down into the water phase
- 25 containing the water-soluble components and with a temperature set at 40-45°C. The distribution of the oil phase with subsequent dissolution in the water phase, the actual solubilising process, must take place under constant stirring and at a suitably calm pace.

30

The mixing procedure

The oil phase (code A): The auxiliary material 05 is added one at a time and in the given sequence, the vitamins 01, 02 and 03 and the antioxidant 04. The components are preheated in order to achieve a suitable consistency for handling. Mixing is effected to uniformity. Hereafter, the mixture will continue to be homogeneous.

The water phase I (code B): Measured amounts of water 15 (preheated to approx. 45°C) are mixed with 14, in which 13, 12, 11 and 10 are dissolved in this order. Thereafter, 08 is added to the mixture (insoluble in water at this process stage). When the material appears to be distributed uniformly in the water phase, the base 07 is added. Upon conclusion of the dissolution of 08, the acid 09 is added and thereafter 06, 05, 04, 03 and 02 one at a time and in this order. The product appears as a slightly opalescent liquid.

Solubilising phase (A \Rightarrow B): The oil phase A is now pumped in a thin and continuous stream down into the water phase I, which is held in constant movement by the slowly-moving stirring unit. Mixing is effected to uniformity.

Completion (C \Rightarrow (A + B)) Phytase (01), performed under water phase II, is mixed in, after which 02 and finally 03 are dissolved. The preparation is now ready for use.

In certain cases, the components 02 and 03 can be added in water phase I.

Comments on the formulation

5 The phytase must be included as a part of the vitamin mixture. This gives rise to the undesired effect that the content of the otherwise water-soluble sodium riboflavin phosphate, when it exists above a certain concentration, is precipitated as riboflavin. The process, which is irreversible under the conditions determined here, will render the finished product useless. The speed at which the precipitation takes place depends first and foremost on the concentration of said components.

10

Urea (carbamide) is used here as a solubility-promoting material for riboflavin and is optimised for the respective formulation (model). With dosing of riboflavin below a certain level, it is not always necessary to add urea, in that it can be sufficient here with the declared amount of nicotinamide (= niacinamide). It is otherwise well-known that this vitamin of the B-group similarly possesses solubility-promoting characteristics. When the use of urea is found in the actual context, it will form part with 2-10% related to weight of the total product.

15

20 If folic acid is included in a formulation, this is handled in the same way as riboflavin. The material is normally insoluble in water with neutral pH, but can be dissolved in basic solutions where, however, it is chemically unstable. Both nicotinamide and urea serve to promote solubility.

25 Urea has been selected for the reason that it exists in an acceptable degree of purity, and at the same time it is found to be reasonably cheap for the purpose. Moreover, no negative stability has been shown with regard to the remaining active material contents, and in the stated concentrations it must be considered to be harmless for internal use.

30

Liquid vitamin mixture, sprayed on after pelleting.

In the following discussion of the results from the spraying of liquid vitamin premix on cooled feed pellets, the tests with broilers have been omitted, the reason being that the liquid vitamin premix in these tests was introduced via drinking water. However, technically and from the point of view of nutrition, there is nothing to prevent the spraying of liquid vitamin premixes on poultry feeds and all other pelleted types of feeds.

Preliminary investigations gave rise to a great deal of conjecture concerning the shown vitamin loss in the production of animal feeds, and therefore it was decided to examine whether feed pellets could be sprayed with liquid vitamin premixes after cooling with a better recovery result – and herewith a better correlation between the added vitamin and the vitamin supplied to the animals. Similarly, it was decided to carry out production tests with decreasing dosage of liquid vitamin premix compared with dry vitamin premix, the object being to determine a possible minimum dosage where the animals reacted via their feedstuff utilisation or growth.

In the trial, the highest dosing of liquid premix corresponded to the dosing of dry vitamin premix to the control feed (index 100 corresponding to standard). The dosing of vitamin to the 4 remaining trials mixtures was reduced to following index: 85, 70, 55 and 40. The only difference in the control and trial feed was the premix type and the method of addition.

All mixtures were dosed with 2 kg. per ton feedstuff.

Table 1. Feeds for porkers. Recovery percentages for vitamins in premixes and pellets at production.

| | Dry premi x | Feed pellets with dry premix | Liquid premix | Feed pellets with liquid premix |
|-------------------------|-------------------|---------------------------------|---------------|------------------------------------|
| Vitamin-A | 79 | 58 | 83 | 77 |
| Vitamin-E | 92 | 68 | 103 | 85 |
| Vitamin-K ₃ | 94 | 4 | 82 | 32 |
| Vitamin-B ₁ | 60 | 39 | 70 | 82 |
| Vitamin-B ₂ | 93 | 74/12 | 101 | 226/72 |
| Vitamin-B ₆ | 67 | 33 | 91 | 71 |
| Vitamin-B ₁₂ | 42 | 18 | 82 | 63 |
| Niacin | 91 | 48 | 97 | 90 |
| Pantothenic acid | 81 | 57 | 98 | - |
| Biotin | 94 | 40/-19 | 95 | 132/53 |

- 5 From Table 1 it will be seen that the recovery percentages for vitamins sprayed on the surface of cooled feed pellets are very much higher than for dry vitamins which have gone through the feed production process. However, it must also be ascertained that the recovery percentages lie below the sprayed-on amounts, with great variation from vitamin to vitamin. The
- 10 investigation does not clarify the question of whether there is an actual vitamin loss due to chemical reactions upon the contact of the vitamins with the surface of the pellets, or whether an analysis problem is involved.

- 15 The vitamins K₃, B₁₂ and biotin had very low recovery percentages after dry addition. With liquid spraying, K₃ and biotin still have low recovery percentages, but nevertheless the recovery is 20 to 25 percentage points higher. For vitamin B₁₂, the recovery is 30 to 40 percentage points higher.

- For vitamin B₁, the results indicate a recovery of around 80 percent, which is 35 to 40 percentage points higher than with dry vitamins. The spraying-on of vitamin B₂, B₆ and niacin shows recovery percentages which are 30 to 40 percentage points higher than with the use of dry vitamins. The vitamins A and E have always attracted the greatest interest in analytical post-control of the vitamin contents of the feed mixture – the average results of the present tests show approximately 20 percentage points better recovery for sprayed vitamin A and approx. 15 percentage points for vitamin E.
- 10 The improved recovery percentages for the vitamins after spraying are naturally of importance for the animals' vitamin supply, and they are not without significance in relation to the feed control authorities. However, the most important acknowledgement is that the vitamin dosing by liquid spraying can be reduced without reducing the animals' supply of the added vitamins.

The storage stability

- 20 The recovery percentages for vitamins added via dry vitamin premixes or sprayed on cooled pellets immediately after production can not stand alone – the storage durability of the vitamins is of great importance both for the feed industry and the animal breeder.
- 25 To follow up on this, samples were taken of a number of the tested feed portions after storage for 2, 4 and 6 weeks in a feed silo. The relatively short storage time is considered to be appropriate for most of the feed mixtures used in modern animal breeding.

Table 2: Storage stability of vitamins in feed pellets. Percentage recovery after pelleting and 6 weeks storage in relation to meal product (natural vitamins) or declared additives (dry and liquid premix).

Index 100 = standard. Average of 4 productions.

| | Feed pellets | | Feed pellets with dry pre-mix added, Index 100 | | Feed pellets with liquid premix added | |
|---------------------------------|------------------|---------|--|---------|---------------------------------------|---------|
| | Natural vitamins | | | | Index 100 | |
| | Production | 6 weeks | Production | 6 weeks | Production | 6 weeks |
| Vitamin-A, i.u./g | 280/100 | 280/100 | 58 | 59 | 79 | 65 |
| Vitamin-E, mg/kg | 112 | 101 | 68 | 65 | 79 | 96 |
| Vitamin-K ₃ , mg/kg | 120 | 44 | 4 | 0/23 | 48 | 4 |
| Vitamin-B ₁ , mg/kg | 91 | 108 | 51 | 36 | 84 | 25 |
| Vitamin-B ₂ , mg/kg | 114 | 117 | 43 | 38 | 67 | 95 |
| Vitamin-B ₆ , mg/kg | 130 | 88 | 33 | 51 | 82 | 88 |
| Vitamin-B ₁₂ , mg/kg | 142 | 98 | 17 | 30 | 76 | 50 |
| Niacin, mg/kg | 102 | 103 | 48 | 55 | 84 | 67 |
| Pantothenic acid mg/kg | 92 | 88 | 57 | 68 | - | - |
| Biotin, mg/kg | 94 | 101 | 20 | 25 | 50 | 75 |
| | | | | | 13/131 | 0 |

Table 2 shows recovery percentages for vitamins at production and after storage for 6 weeks. For feed pellets which only contain natural vitamins, the recovery in pellets is set in relation to the contents in the meal product. For the remaining mixtures, the recovery is expressed in relation to the guaranteed addition. Recovery after 2 and 4 weeks' storage is omitted out of regard for space, and because they do not influence the general tendency.

In general it can be ascertained that all naturally occurring vitamins would appear to have a greater storage stability.

Vitamin A shows a slight storage loss after spraying, but the loss is considerably less than expected. For the vitamins E, B₂ and B₆, there is no systematic storage loss, and pantothenic acid added via dry premix and in natural form appears to be very stable. Pantenol applied via liquid premix can also be assumed to be a stable vitamin. The vitamin K₃ retained after liquid application is quickly reduced during storage and it approaches 0 at 4 weeks' storage, but the amounts shown were found upon analysis after 6 weeks. For vitamin B₁ there is great storage stability for natural vitamin. During the storage period, the high recovery level after spraying falls down to the same level as found with dry vitamin addition. For vitamin B₁₂, great difference is seen between the tests, both with regard to level and tendency. However, the amount of vitamin remaining after storage for 6 weeks will be considerably higher after liquid spraying than after dry addition. The result for niacin shows a tendency towards greater loss with liquid spraying than with dry addition, while the result for biotin hardly permits itself to be interpreted due to the great variation in data. A guarded interpretation could be that there is 20 to 30 percent remaining after 6 weeks regardless of the method of application.

The general result of the storage test is that the storage stability remains the same regardless of the method of application. The exceptions are vitamin K₃, B₁ and perhaps B₁₂, which after liquid spraying shows some decrease. However, the contents of these vitamins at the start of the storage test were considerably higher after liquid spraying than with dry addition. If there are to be reasonable amounts of vitamin K₃ and B₁ in the feed after liquid spraying, the feed must be used within 2 to 4 weeks after production.

The tests carried out show a surprisingly great process loss of all vitamins added to the feed via dry vitamin premix. The lacking vitamin recovery starts already after admixture with the meal product. A number of vitamins lose further activity during expansion, and there is a tendency towards continued loss during the subsequent pelleting. In feed pellets, the average recovery for added vitamin E and pantothenic acid is highest with approx. 70 percent, while the recovery percentages for the remaining vitamins lie a good 50 to approx. 25 percent down. Vitamin K₃ is particular in that it more or less disappears.

The average recovery percentages for vitamins sprayed on feed pellets after cooling lies from 15 to 60 percentage points higher than for vitamins added in the form of dry vitamin premix (table 1).

Since at the same time the storage stability for the vitamins with up to 4 weeks' storage of the feed mixtures is generally the same regardless of the method of application, the animals are provided with considerably higher amounts of vitamin at the same vitamin dosing to the feed when the vitamins are sprayed on as liquid premix.

The content of natural B vitamins proves to be extremely stable during processing, and even with a tendency towards increasing content during the feed production process. With the content level taken into con-

sideration, it is not curious that a feed mixture seldom or never falls at the post-control for the content of B vitamins. The same applies to a considerable extent regarding vitamin E. Vitamin A and especially vitamin K₃ do not have this natural back-up, and since both vitamins in synthetic form are somewhat or very sensitive to processing, it is obvious that post-control here will reveal many failures.

The recovery percentages in pellets for vitamins added via dry vitamin premixes show that there is a very poor correlation between the declared vitamin content (added) in the feed mixtures and that amount which reaches the animals. The relationship is considerably better when the pellets are sprayed with vitamins. In light of this, it was natural to carry out an investigation into whether the animals in production tests reacted to reduced vitamin addition via liquid premix sprayed on the cooled feed pellets.

The results of the production tests showed that porkers did not react to a reduction of vitamin allocation via liquid spraying of 60 percent of the prescribed dry vitamin addition.

The tests performed show that a considerably higher percentage of the added vitamins reach the animals when the vitamin premix is sprayed on the finished feed or dosed via the drinking water. Consequently, due to the elimination of the process loss, the animals' vitamin requirements can be covered with a smaller addition of vitamins after pelleting.

The concept involves the addition of phytase enzyme to expanded/pelleted poultry feeds and pig feeds. When the enzyme is sprayed on cooled feed pellets together with the vitamin premix, the full enzyme activity is retained, which gives rise to the release of 60 to 70 percent of the organically-bound phosphor in vegetable feed raw materials. Furthermore, smaller amounts of amino acids, carbohydrates and micro-minerals are released. As a con-

sequence of the use of enzymes and reduced dosing of mineral phosphate, it is expected that the concentration of phosphor in the manure and the discharge to the environment can be reduced by 20 to 25 percent.

- 5 The micro-minerals in a liquid micro-mineral mixture are 100 percent dissolved, the result being that the accessibility to and the absorption by the animals is greater than with the use of traditional salts. As a consequence, the dosing of micro-minerals to industrially-produced feeds can be reduced, with subsequently less discharge to the environment.

10

The new products can be delivered in packaging which can be handled by truck, and the products are conveyed from the packaging to the spraying equipment in the feed mill through closed piping systems. This results in considerable advantages for the animal feed industry, both with regard to

- 15 handling as well as the working environment.

AUXILIARY REQUEST

1. Method for the production of animal feed pellets with the addition of a premix, c h a r a c t e r i z e d in that the premix is a vitamin premix comprising fat/oil- and water-soluble vitamins, that the surface of the feed pellets is sprayed with the vitamin premix, and in that the feed pellets are subjected to cooling before being sprayed said temperature being equal or less than 50°C and that the vitamin premix also comprises a phytase enzyme dissolved in said vitamin premix and that after being sprayed the feed pellets are collected in a container.
2. Method according to claim 1, c h a r a c t e r i z e d in that the vitamin premix is formulated as a function of animal species.
3. Method according to any of the foregoing claims, c h a r a c t e r i z e d in that the feed pellets pass a rotor-spray/rotor nozzle when being sprayed.
4. Method according to any of the foregoing claims, c h a r a c t e r i z e d in that the feed pellets are also sprayed with a solution comprising minerals.
5. Method according to any of the foregoing claims, c h a r a c t e r i z e d in that the vitamin premix also comprises amino acids dissolved in said vitamin premix.
6. Method according to any of the foregoing claims, c h a r a c t e r i z e d in that the vitamin premix also comprises digestibility-promoting enzymes dissolved in said vitamin premix.
7. Method for the mixing a vitamin premix comprising fat/oil-soluble vitamins, c h a r a c t e r i z e d in that the premix also comprises water-soluble vitamins, that the water phase comprises propylene glycol and EDTA and

nicotinamide, after which a B₂ vitamin is subsequently added such as riboflavin and thereafter sodium hydrochloride (NaOH).

5 8. Method according to claim 7, characterized in that carbamide/urea is added before the addition of the B₂ vitamin.

9. Method according to claim 7 and 8, characterized in that hydrochloric acid (HCl) is also added, and that further B vitamins are subsequently added, mainly biotin and pyridoxine hydrochloride.

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10. Method according to claim 7, 8 and 9, characterized in that the oil phase comprises A, D and E vitamins, a solubilisator and also antioxidants, the mixing of which is carried out at a temperature interval of around 50-70°, preferably at around 60°.

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11. Method according to claims 7-10, characterized in that the oil phase and the water phase are mixed together while being stirred, and that the temperature of the water phase is 35-45°C.

20 12. Method according to claims 7-11, characterized in that a phytase enzyme is added to the vitamin premix, said premix preferably having a temperature of 20-30°C.

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U.S. DEPARTMENT OF COMMERCE
PATENT AND TRADEMARK OFFICE

DECLARATION AND POWER OF ATTORNEY

ATTORNEY'S DOCKET NO.
12085/1

As a below named inventor, I hereby declare that:

My residence, post office address, and citizenship are as stated below next to my name,

I believe I am an original, first, and sole inventor of the subject matter that is claimed and for which a patent is sought on the invention entitled **METHOD FOR THE PRODUCTION OF FEED PELLETS, MIX OF A PREMIX AND VITAMIN PREMIX**, the specification of which was filed as U.S. Serial No. 09/831,243, which is the U.S. National Phase of International Application No. PCT/DK99/00559, filed October 15, 1999.

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims.

I acknowledge the duty to disclose information which is material to the examination of this application in accordance with Title 37, Code of Federal Regulations, § 1.56(a).

PRIOR FOREIGN APPLICATION(S)

I hereby claim foreign priority benefits under Title 35, United States Code, § 119(a)-(d) or §365(b) of any foreign application(s) for patent or inventor's certificate, or §365(a) of any PCT International application which designated at least one country other than the United States, listed below and have also identified below any foreign application for patent or inventor's certificate or PCT International application having a filing date before that of the application on which priority is claimed:

| COUNTRY | APPLICATION NUMBER | DATE OF FILING (day, month, year) | DATE OF ISSUE (day, month, year) | PRIORITY CLAIMED UNDER 35 U.S.C. § 119 |
|---------|--------------------|--------------------------------------|-------------------------------------|---|
| Denmark | PA 1998 01422 | 4 November 1998 | | Yes |
| Denmark | PA 1999 00055 | 18 January 1999 | | Yes |

POWER OF ATTORNEY: As a named inventor, I hereby appoint the following attorneys:

Stuart J. Sinder (25,377)
Christine M. Wilkes (37,967)

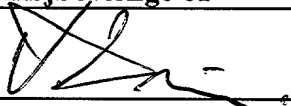
SEND CORRESPONDENCE, AND DIRECT TELEPHONE CALLS TO:

KENYON & KENYON
One Broadway
New York, New York 10004
(212) 425-7200 (phone)
(212) 425-5288 (facsimile)



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PATENT TRADEMARK OFFICE

I declare that all statements made herein of my own knowledge are true and all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under § 1001 of Title 18 of the United States Code and that such willful statements may jeopardize the validity of the application or any patent issuing thereon.

| | | | |
|---|--|---|---|
| FULL NAME OF INVENTOR | FAMILY NAME DUER | FIRST GIVEN NAME Victor | SECOND GIVEN NAME |
| RESIDENCE & CITIZENSHIP | CITY DK-3500 Værløse | STATE OR FOREIGN COUNTRY Denmark DKX | COUNTRY OF CITIZENSHIP Denmark |
| POST OFFICE ADDRESS | POST OFFICE ADDRESS Højbovænge 61 | CITY Værløse | STATE & ZIP CODE/COUNTRY Denmark |
| Signature  | | Date 17 June 2001 | |